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MOISTURE RELATIONS OF BANDED INGREDIENTS IN AN ILLINOIS COAL

BY

O. W. REES, G. W. LAND, AND F. H. REED

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By

O. W. Rees, G. W. Land, and F. H. Reed

ABSTRACT

The moisture-humidity relations of the banded ingredients vary in relation to each other and to the whole coal. The moisture-humidity curve for vitrain lies above that of whole coal, that of clarain very near that of whole coal, that of durain below that of whole coal, and that of fusain below all others up to about 96 per cent humidity, at which value it rises sharply well above all others.

The moisture values of the whole coal at different humidities appear to be weighted composites of the moisture values of the component ingredients. The equilibration and calculated pore-size data appear to correlate well with the capillary theory for the occurrence of moisture in coal. The variations in the moisture relations of whole coal and its component banded ingredients as shown by studies on this one coal have a distinct bearing on moisture in commercially prepared coals.

In studying the applicability of the Stansfield-Gilbart equilibration method (*8*) to the determination of bed moisture in Illinois coal for classification by rank (*2*) data were obtained which, when plotted, gave irregular curves that could not be satisfactorily extrapolated (*7*). The irregularities were believed to be caused by differences in the moisture-humidity relations of the petrographic constituents or banded ingredients of the coal. Accordingly, a study of these relations was started. This is a report of equilibration data obtained for whole coal and for the four component bands, fusain, durain, clarain, and vitrain, obtained from one mine. Two sets of samples were taken from the same mine at different times, and moisture studies were made on each set.

SAMPLES AND PROCEDURE

Coal No. 6 from Franklin County was used in this study. Samples of whole coal were obtained from freshly exposed faces. Portions of the samples were crushed in the mine to —14 mesh and then were placed in glass jars under distilled water for storage; the water had been boiled to expel dissolved gases, particularly oxygen and carbon dioxide. Other portions of the samples were placed in ordinary airtight cans in the mine and were later used for chemical analysis and equilibration. The samples of the four banded ingredients were obtained by hand picking and were crushed to —14 mesh in the mine. Some portions were sealed in glass jars under water, and other portions were placed in airtight sample cans for immediate chemical analysis and equilibration. The two sets of samples were handled in the same way, the coal stored under water being later subjected to equilibration. The whole coal of the first set was stored under water 38 days before equilibration, the fusain 15 days, the clarain 19 days, the vitrain 23 days, and the durain 27 days. The whole coal of the second set was stored 27 days, fusain and clarain 16 days, and vitrain and durain 24 days before equilibration. Of the samples brought to the laboratory for immediate equilibration, whole coal and clarain were equilibrated first, vitrain and fusain next, and durain last. Equilibrations of whole coal and clarain were started 2 days after sampling, equilibrations of vitrain and fusain 4 days after sampling, and equilibrations of durain 6 days after sampling. These latter samples of vitrain, fusain and durain were stored at 90 per cent relative humidity at 30° C. until equilibrated.

Proximate analyses were made on all samples according to A. S. T. M. procedures (*1*). Five-gram samples of the whole coal and the four banded ingredients were equili-

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Table 1.—Chemical Analyses

Lab No.	Description	Moisture as Received, %	Ash[a], %	Volatile Matter[a], %	Fixed Carbon[a], %	Total Sulfur[a], %	Calorific Value[a], B. t.u.
C-2138	Coal	8.9	8.5	37.5	54.0	1.9	13,161
C-2139	Fusain	23.0	6.6	10.4	83.0	4.0	13,849
C-2140	Durain	6.1	14.8	37.5	47.7	1.1	12,368
C-2141	Clarain	5.6	3.8	41.0	55.2	1.2	14,000
C-2142	Vitrain	10.5	2.7	36.2	61.1	1.3	13,996
C-2291	Coal						
	1[b]	9.0	8.6	37.8	53.6	1.6	13,261
	2[c]		8.2	37.4	54.4	1.6	13,344
C-2292	Fusain						
	1[b]	19.0	4.6	11.3	84.1	1.9	14,206
	2[c]		4.0	11.2	84.8	1.9	14,396
C-2294	Durain						
	1[b]	6.5	14.9	36.2	48.9	1.2	12,340
	2[c]		15.4	36.9	47.7	1.1	12,380
C-2293	Clarain						
	1[b]	8.3	3.8	41.0	55.2	1.5	13,965
	2[c]		3.6	41.0	55.4	1.5	13,961
C-2295	Vitrain						
	1[b]	9.3	3.2	37.8	59.0	1.2	13,914
	2[c]		2.8	37.6	59.6	1.2	13,999

[a]On a dry basis. [b]Not water-stored. [c]Water-stored.

brated at eight different humidities ranging from 11.2 to 96.7 per cent. The equilibrations were made in desiccators immersed in a waterbath thermostat controlled at 30° ± 0.05° C. with evacuation every 12 hours, using saturated salt solutions to produce desired humidities as previously described (*7*, *8*). After equilibration for 48 hours the samples were removed from the desiccators, and residual moisture was determined in a vacuum oven by heating at 105° C. for 3 hours in a nitrogen atmosphere under a pressure of 3 inches (7.6 cm.) of mercury. Equilibrations were made on the whole coal before and after water storage, on banded-ingredient samples after water storage in the first series, and on all samples of the second series, both before and after water storage. Equilibrations and moisture determinations were made in duplicate. For the first set of samples on which forty-eight determinations were made, the average numerical deviation between duplicates was 0.05 per cent with a maximum deviation of 0.93 per cent. For the second set of samples on which eighty determinations were made, the average numerical deviation between duplicates was 0.06 per cent with a maximum deviation of 2.28 per cent. The two maximum deviations noted were for fusain samples equilibrated at 96.7 per cent humidity. The maximum deviations between duplicates exclusive of these two samples were 0.24 and 0.10 per cent for the first and second sets of samples, respectively.

RESULTS

Table 1 presents proximate analyses for for the samples studied. The moisture values reported in this table were obtained by the usual A. S. T. M. procedure, including preliminary air-drying. Table 2 gives the equilibration data for all samples. Data for the first set are presented graphically in figure 1. Figure 2 shows equilibration data for samples of the second set which were not water-stored. Figure 3 presents data for equilibrations of samples of the second set which were water-stored.

Table 2 and figures 1, 2, and 3 show that the moisture-humidity relations of the banded ingredients vary widely among themselves. In addition, curves for three of the four banded ingredients vary in position or in both position and shape from those of the whole coal. The differences in shape of the curves for whole coal, water-stored and not water-stored, are discussed later. The fusain curve lies well below those for all other samples until a high humidity is reached. Up to about 90 per cent humidity fusain does not take up much moisture, but at 96.7 per cent it does take up a large amount of moisture exceeding all other samples. On the other hand, the vitrain curve lies above all others except at 96.7 per cent humidity, where fusain exceeds it. This curve is, in general, the same shape as that for the whole coal but differs in its higher position. The curve for clarain

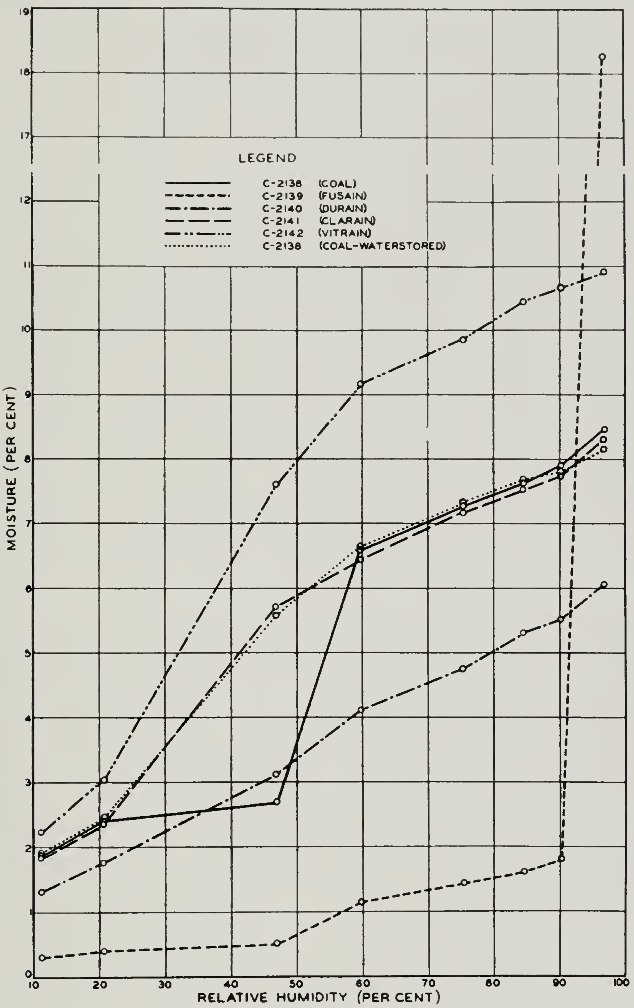

Fig. 1. Moisture-Humidity Curves for First Set of Samples, Water-Stored

is very much like that for the whole coal; and although the curve for durain has the same general shape, it lies considerably below that of the whole coal. Both sets of curves show the same general relations but differ in the magnitude of variations.

In previous work (7) reported by the authors in which the application of the equilibration method for determining bed moisture in Illinois coals was studied, irregular curves were obtained. At that time it was suggested that these irregularities were significant and that it was not proper to draw smooth curves for extrapolation. We believe that the variations in the moisture-humidity relations for the banded ingredients making up the whole coal, as reported here, confirm the impropriety of drawing smooth curves for extrapolation.

In order to obtain some information on the effect of water storage, the whole coal (C-2138) was equilibrated both before and after water storage. Table 2 and figure 1 show that the two agree closely, with the exception that the moisture value at 46.9 per cent humidity was considerably lower in the sample which was not water-stored. To check this deviation and further establish the validity of equilibration data obtained on water-stored samples, the second set of samples was secured from the same mine and as nearly as possible at the same place in the mine. Again the banded-ingredient samples were hand-picked in the mine. Equilibrations

Table 2.—Equilibration Data—Per Cent Moisture

Lab. No.	Description	% Relative Humidity: 11.2	20.6	46.9	59.8	75.4	84.4	90.1	96.7
C-2138	Coal								
	1[a]	1.9	2.4	2.7	6.6	7.3	7.6	7.9	8.5
	2[b]	1.9	2.5	5.6	6.6	7.3	7.7	7.8	8.2
C-2139	Fusain	0.33	0.43	0.53	1.2	1.5	1.6	1.8	18.3
C-2140	Durain	1.3	1.8	3.1	4.1	4.9	5.3	5.5	6.1
C-2141	Clarain	1.8	2.4	5.7	6.5	7.2	7.5	7.8	8.3
C-2142	Vitrain	2.2	3.1	7.6	9.2	9.9	10.5	10.7	10.9
C-2291	Coal								
	1[a]	1.8	2.6	5.5	7.2	8.3	8.5	8.8	9.2
	2[b]	1.9	2.7	5.3	7.3	8.4	8.7	9.0	9.3
C-2292	Fusain								
	1[a]	0.36	0.58	1.0	1.8	3.1	3.6	4.7	11.2
	2[b]	0.43	0.58	0.90	1.3	1.8	2.0	2.2	14.3
C-2294	Durain								
	1[a]	1.5	2.0	3.4	4.4	5.1	5.4	5.6	6.3
	2[b]	1.4	2.0	3.7	4.3	5.1	5.5	5.7	6.3
C-2293	Clarain								
	1[a]	1.9	2.8	5.5	7.0	7.8	8.1	8.3	8.6
	2[b]	2.1	2.7	5.0	6.8	7.6	8.0	8.2	8.6
C-2295	Vitrain								
	1[a]	2.2	3.2	6.3	7.9	8.6	9.0	9.2	9.5
	2[b]	2.2	3.1	7.0	8.0	8.8	9.1	9.3	9.8

[a] Not water-stored. [b] Water-stored.

were made on these samples, both with and without water storage. Results shown in table 2 and figures 2 and 3 indicate that water storage does not change the moisture relations of these samples to one another. Moisture-humidity curves for water-stored and unstored whole coal, durain, clarain, and vitrain check closely. The moisture-humidity curve for the sample of fusain which was water-stored lies very near the curve for the unstored sample at the three lowest humidities, is decidedly below it at intermediate humidities, and rises noticeably above it at 96.7 per cent humidity. The deviations in the curves for this fusain sample and for the whole coal of the first set of samples cannot be explained readily and are receiving further study. However, for the comparison of the general moisture characteristics of whole coal and banded-ingredient samples, data obtained on either water-stored or unstored samples appear to be reliable.

A microscopic examination of the —14 mesh whole coal of the second set of samples showed 4.8 per cent fusain, 1.5 per cent durain, 46.7 per cent clarain, and 47.0 per cent vitrain. From the moisture values for these various constituents it should be possible to calculate moisture values for the whole-coal samples equilibrated at various humidities. Table 3 presents such calculated values for the percentage of the total moisture contributed by each band at each humidity. This table shows that calculated moisture values for the whole coal check well with the determined values. It is of interest to note that fusain contributes only 1.0 per cent of the total moisture of the whole coal at 11.2 per cent humidity and 5.9 per cent at 96.7 per cent humidity. The water-stored sample of fusain contributed a slightly higher proportion (6.7 per cent) of the total moisture at this humidity. Throughout the range of humidity studied for both water-stored and unstored samples, durain accounts for about 1 per cent, clarain for approximately 45 per cent, and vitrain for approximately 52 per cent of the total. Values for clarain samples which had been previously water-stored were somewhat more erratic than values for the unstored samples.

MOISTURE CONTENT *vs.* PORE SIZE

The data provided by these equilibration studies permit us to examine the theoretical implications inherent in the assumption that the moisture in coal is present in pores. Lavine and others (*4, 5*) applied the Thomson equation (*9*) to moisture-humidity data for calculating the sizes and distribution of pores in lignite. Lowry and Hulett (*6*) used Anderson's formula (*3*) in calculating sizes of capillaries in charcoal in connection with

TABLE 3.—MOISTURE VALUES CALCULATED FROM EQUILIBRATION DATA AND PETROGRAPHIC ANALYSIS

Humidity, %	C-2292 Fusain				C-2294 Durain				C-2293 Clarain			
	Weighted H_2O, %		% total H_2O in whole coal		Weighted H_2O, %		% total H_2O in whole coal		Weighted H_2O, %		% total H_2O in whole coal	
	1[a]	2[b]	1[a]	2[b]	1[a]	2[b]	1[a]	2[b]	1[a]	2[b]	1[a]	2[b]
11.2	0.02	0.02	1.0	1.0	0.02	0.02	1.0	1.0	0.89	0.98	45.4	47.8
20.6	0.03	0.03	1.0	1.1	0.03	0.03	1.0	1.1	0.31	1.26	45.6	45.3
46.9	0.05	0.04	0.9	0.7	0.05	0.06	0.9	1.0	2.57	2.34	45.6	41.6
59.8	0.09	0.06	1.3	0.8	0.07	0.06	1.0	0.8	3.27	3.18	45.8	45.0
75.4	0.15	0.09	1.9	1.1	0.08	0.08	1.0	1.0	3.64	3.55	46.0	45.2
84.4	0.17	0.10	2.1	1.2	0.08	0.08	1.0	1.0	3.78	3.74	45.8	45.6
90.1	0.23	0.11	2.7	1.3	0.08	0.09	0.9	1.1	3.88	3.83	45.6	45.6
96.7	0.54	0.67	5.9	6.7	0.09	0.09	1.0	0.9	4.02	4.02	44.1	40.0

Humidity, %	C-2295 Vitrain				% Moisture in Whole Coal C-2291			
	Weighted H_2O, %		% total H_2O in whole coal		Calculated		Determined	
	1[a]	2[b]	1[a]	2[b]	1[a]	2[b]	1[a]	2[b]
11.2	1.03	1.03	52.6	50.2	1.96	2.05	1.8	1.9
20.6	1.50	1.46	52.3	52.5	2.87	2.78	2.6	2.7
46.9	2.96	3.29	52.6	57.4	5.63	5.73	5.5	5.3
59.8	3.71	3.76	52.0	53.3	7.14	7.06	7.2	7.3
75.4	4.04	4.14	51.1	52.7	7.91	7.86	8.3	8.4
84.4	4.23	4.28	51.2	52.2	8.26	8.20	8.5	8.7
90.1	4.32	4.37	50.8	52.0	8.51	8.40	8.8	9.0
96.7	4.47	5.26	49.0	52.4	9.12	10.04	9.2	9.3

[a] Not water-stored.
[b] Water-stored.

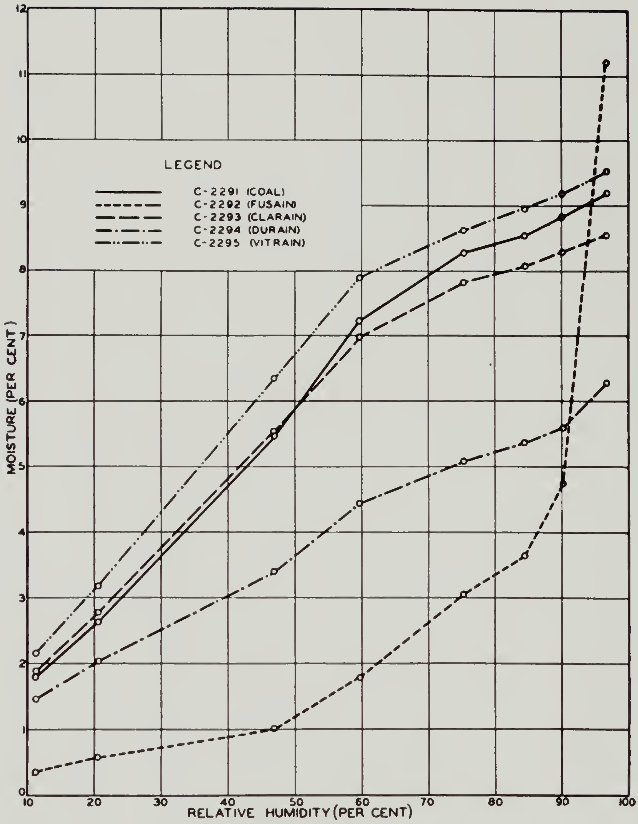

FIG. 2. Moisture-Humidity Curves for Second Set of Samples, Not Water-Stored

studies of moisture in charcoal. Such calculations are based on the fact that a change in the radius of curvature of liquid in a capillary results in a change of the vapor pressure of that liquid. The Thomson equation derivation assumes a spherical meniscus, and to obtain this the adsorption layer must be thin in respect to the diameter and the capillary walls must be wet by the liquid in them. Applying this equation to the data furnished by equilibration studies as given in Table 2, values for the percentage of moisture in various ranges of pore size were calculated for the samples studied and listed in Tables 4 and 5. These tables show that the banded ingredients vary considerably in the amount of moisture held in different size pores. For example, in fusain we find that the

TABLE 4.—CALCULATED MOISTURE HELD IN VARIOUS PORE SIZES UP TO 96.7 PER CENT RELATIVE HUMIDITY BY SAMPLES OF FIRST SET (ALL WATER-STORED)

	Whole Coal C-2138		Fusain C-2139		Durain C-2140		Clarain C-2141		Vitrain C-2142	
Radius Range, Cm. × 10^{-7}	Moisture held, %	Cumulative, %	Moisture held, %	Cumulative, %	Moisture held, %	Cumulative, %	Moisture held, %	Cumulative, %	Moisture held, %	Cumulative, %
9.80–30.60	4.9	4.9	90.2	90.2	9.8	9.8	6.0	6.0	1.8	1.8
6.02–9.80	1.2	6.1	1.1	91.3	3.3	13.1	3.6	9.6	1.8	3.6
3.61–6.02	4.9	11.0	0.5	91.8	6.6	19.7	3.6	13.2	5.5	9.1
1.99–3.61	8.5	19.5	1.6	93.4	13.1	32.8	8.4	21.6	6.4	15.5
1.35–1.99	12.2	31.7	3.7	97.1	16.4	49.2	9.6	31.2	14.7	30.2
0.65–1.35	37.8	69.5	0.5	97.6	21.3	70.5	39.8	71.0	41.3	71.5
0.47–0.65	7.3	76.8	0.5	98.1	8.2	78.7	7.2	78.2	8.3	79.8
0.0–0.47	23.2	100.0	1.9	100.0	21.3	100.0	21.8	100.0	20.2	100.0

largest amount of moisture is held in large pores of the size range 9.80–30.60 $\times 10^{-7}$ cm. Durain, clarain, and vitrain have the largest amount of moisture in pores of the size range 0.65–1.35 $\times 10^{-7}$ cm. More than 90 per cent of the total amount of moisture held by fusain at 96.7 per cent humidity is held in pores of the size range 1.35–1.99 $\times 10^{-7}$ cm. and larger. No other banded ingredient approaches this, durain having only about 45 per cent of its moisture in this range, clarain only about 35 per cent, and vitrain only about 30 per cent. Water storage appears to increase the relative amount of the moisture which is held in larger pores of fusain. No particularly noticeable effects are apparent in the other banded ingredients.

The authors make no claim of having discovered experimentally that pore spaces exist in coal. The correlation is presented as a matter of probable interest and one that is being further investigated in this laboratory.

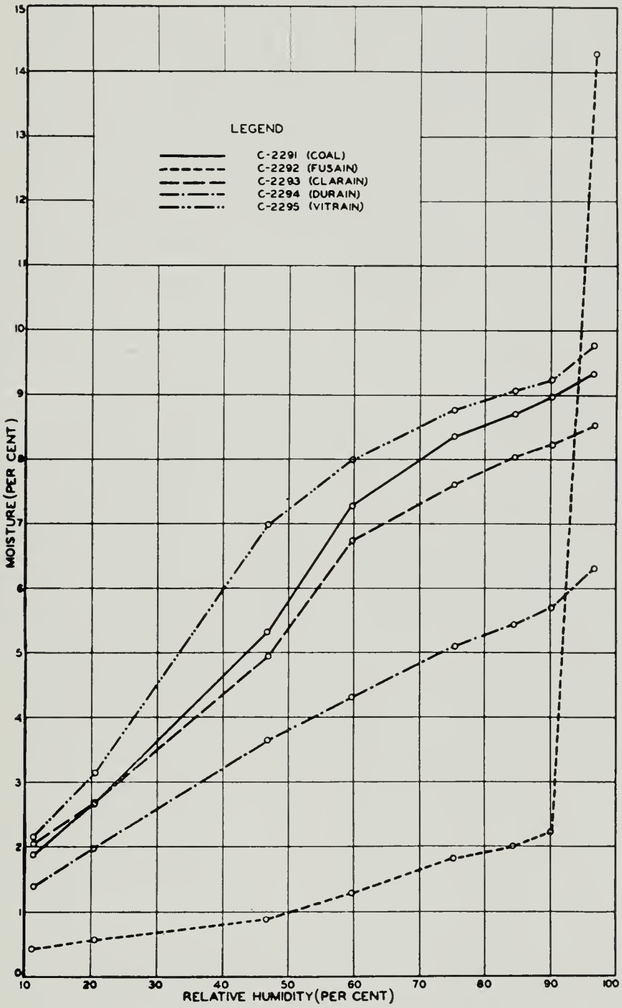

FIG. 3. Moisture-Humidity Curves for Second Set of Samples, Water-Stored

PRACTICAL SIGNIFICANCE

Aside from the theoretical considerations involved in the manner of occurrence of moisture in coal and in the banded ingredients as demonstrated by the equilibration data, there are certain important practical consequences. In the industrial processing of coal the banded ingredients are concen-

Table 5.—Calculated Moisture Held in Various Pore Sizes up to 96.7 Per Cent Relative Humidity by Samples of Second Set

	Whole Coal C-2291		Fusain C-2292		Durain C-2294		Clarain C-2293		Vitrain C-2295	
Radius Range, Cm. × 10^{-7}	Moisture held, %	Cumulative, %	Moisture held, %	Cumulative, %	Moisture held, %	Cumulative, %	Moisture held, %	Cumulative, %	Moisture held, %	Cumulative, %
9.80–30.60	4.3[a]	4.3	58.0	58.0	11.1	11.1	3.5	3.5	3.2	3.2
	3.2[b]	3.2	84.6	84.6	9.5	9.5	4.7	4.7	5.1	5.1
6.02–9.80	3.3	7.6	9.8	67.8	3.2	14.3	2.3	5.8	2.1	5.3
	3.2	6.4	1.4	86.0	3.2	12.7	2.3	7.0	2.0	7.1
3.61–6.02	2.2	9.8	4.5	72.3	4.8	19.1	3.5	9.3	4.2	9.5
	3.2	9.6	1.4	87.4	6.3	19.0	4.7	11.7	3.1	10.2
1.99–3.61	12.0	21.8	11.6	83.9	11.1	30.2	9.3	18.6	7.4	16.9
	11.8	21.4	3.5	90.9	12.7	31.7	9.3	21.0	8.2	18.4
1.35–1.99	18.5	40.3	7.1	91.0	15.9	46.1	17.4	36.0	16.8	33.7
	21.5	42.9	2.8	93.7	9.5	41.2	20.9	41.9	10.2	28.6
0.65–1.35	31.5	71.8	3.8	94.8	22.2	68.3	31.4	67.4	32.6	66.3
	28.0	70.9	2.2	95.9	27.0	68.2	26.7	68.6	39.8	68.4
0.47–0.65	8.7	80.5	2.0	96.8	7.9	76.2	10.5	77.9	10.5	76.8
	8.6	79.5	1.0	96.9	9.5	77.7	7.0	75.6	9.2	77.6
0.0–0.47	19.5	100.0	3.2	100.0	23.8	100.0	22.1	100.0	23.2	100.0
	20.5	100.0	3.1	100.0	22.3	100.0	24.4	100.0	22.4	100.0

[a] The first value in each case represents samples not water-stored.
[b] The second value represents water-stored samples.

trated. Such concentration may result in a product which has a different moisture-holding capacity from that of the original coal in the mine. For example, if a process is used which results in fusain concentration, the moisture-holding capacity of the product may be considerably lower than that of the original coal when handled at humidities up to 90 per cent, or considerably higher when handled at higher humidities. On the other hand, if the process should concentrate vitrain, the moisture-holding capacity of the product may be higher than that of the unprocessed coal. In this connection the authors recently had the opportunity to compare the moisture-holding capacity of a prepared coal with a face sample from a Pennsylvania mine. It was found that throughout the range of relative humidity from 11.2 to 96.7 per cent the prepared sample had an appreciably higher moisture-holding capacity than the face sample. Petrographic analyses of these two samples showed that the percentage of vitrain was considerably higher in the prepared coal than in the face coal.

As stated before, results reported in this paper were obtained in studies on one Illinois coal only. Further studies are being made to include Illinois coals of higher and lower moisture content.

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